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CREEP AND TENSILE PROPERTIES OF SEVERAL OXIDE-DISPERSION-STRENGTHENED NICKEL-BASE ALLOYS AT 1365 K

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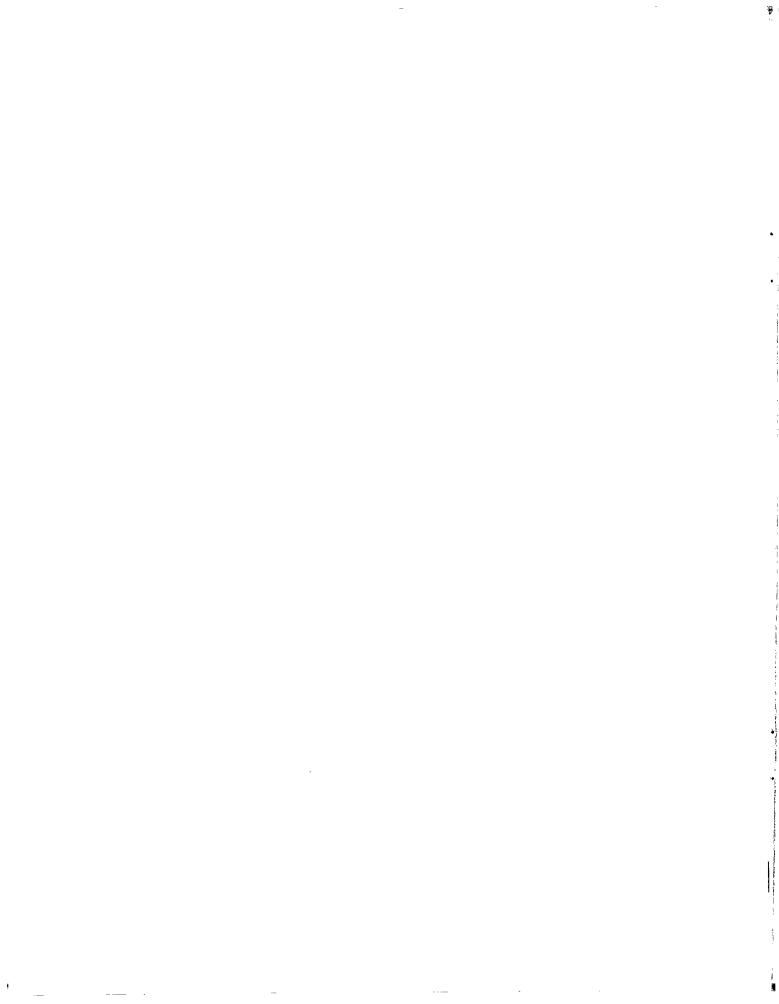
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by J. Daniel Whittenberger

Lewis Research Center

SUMMARY

The tensile properties at room temperature and at 1365 K and the tensile creep properties at low strain rates at 1365 K were measured for several oxide-dispersion-strengthened (ODS) alloys. The alloys examined included ODS Ni, ODS Ni-20Cr, and ODS Ni-16Cr-Al. Metallography of creep-tested, large-grain-size ODS alloys indicated that creep of these alloys is an inhomogeneous process. All alloys appear to possess a threshold stress for creep. This threshold stress is believed to be associated with diffusional creep in the large-grain-size ODS alloys and normal dislocation motion in perfect single-crystal (without transverse low-angle boundaries) ODS alloys. Threshold stresses for large-grain-size ODS Ni-20Cr and Ni-16Cr-Al type alloys are dependent on the grain aspect ratio. Because of the deleterious effect of prior creep on mechanical properties of large-grain-size ODS alloys, it is speculated that the threshold stress may be the design-limiting creep-strength property.

INTRODUCTION

Oxide-dispersion-strengthened (ODS) nickel-base alloys have potential for use in rather demanding elevated-temperature environments, such as aircraft turbine engines. The first generation of commercial ODS nickel alloys, nominally Ni-2ThO₂, exhibited excellent strength in the temperature range of 1255 to 1475 K and over-temperature capacity almost up to the melting point (refs. 1 and 2). Unfortunately, the Ni-2ThO₂ alloys were subject to severe oxidation attack (ref. 3). For improved oxidation resistance, the Ni-20Cr-2ThO₂ type alloys were developed. These alloys possess good elevated-temperature strength and over-temperature capacity (refs. 4 and 5) plus excellent static oxidation resistance (ref. 6); however, the dynamic oxidation resistance at 1255 K and above was low because of the volatization of chromium oxide (refs. 7 and 8). Most recently,

Ni-16Cr-Al (nominally 4 to 6 weight percent aluminum) type ODS alloys have been developed (refs. 9 and 10). These alloys form a protective oxide scale of Al₂O₃ and possess dynamic oxidation resistance as well as other desirable properties (refs. 10 and 11). Ni-16Cr-Al alloys containing either thoria or yttria as the dispersoid have been produced; yttria strengthened alloys are especially attractive as they eliminate the radioactivity problems associated with thoriated alloys.

The high-temperature strength of ODS alloys is due to the presence of a uniform dispersion of fine, inert particles (ref. 12). The particles produce direct strengthening by acting as dislocation barriers; and in polycrystalline ODS alloys, the particles affect the grain size and grain shape, which also influences the mechanical properties (ref. 12). In general, only an elongated grain structure is required for strength parallel to the direction of primary working; however, a large-grain-size, semi-elongated microstructure is probably necessary for reasonable transverse strength (ref. 13). Most theories of strengthening in ODS alloys have considered the consequences of the direct strengthening effect involving interactions between the dispersoid particles and dislocations (ref. 12). However, in creep-type applications, deformation mechanisms involving the grain structure may be more important. For example, recent work by Kane and Ebert (ref. 14) estimated that creep at 1365 K in polycrystalline TD-NiCr (Ni-20Cr-2ThO $_2$) is entirely due to diffusion-controlled grain boundary sliding, that is, diffusional creep (ref. 15). Some microstructural evidence (refs. 16 and 17) of diffusional creep exists for several polycrystalline ODS alloys. In addition, several investigations of ODS alloys have presented data which suggest that creep is not a homogeneous process and that a threshold stress for creep (stress below which creep does not occur) exists (refs. 16 to 19).

This work was undertaken to study the creep behavior at 1365 K of typical ODS Ni, ODS Ni-20Cr, and ODS Ni-16Cr-Al alloys. In general, creep exposures involved low strains (1 percent or less), after a nominal 100 hours of testing. Particular attention was directed toward assessment of threshold stress for creep and examination of microstructures for evidence of diffusional creep and inhomogeneity of creep. In addition, short-term tensile properties at room temperature and at 1365 K were determined.

EXPERIMENTAL PROCEDURE

The nominal compositions of the ODS alloys evaluated are shown in table I. Two alloys were produced by Fansteel, Inc.: TD-Ni in the form of heat-treated (stress-relieved), 1.27-centimeter-diameter bar, and TD-NiCrAl as a steel-canned, 20-centimeter-diameter extrusion billet. DS-NiCr was obtained from Stellite Division of Cabot Corporation in the form of unrecrystallized, 1.9- by 6.4-centimeter bar. Inconel MA-754 was produced by Hunting Alloy, Inc., in the form of heat-treated, 2.8- by 8.3-centimeter bar. The experimental alloy DST-NiCrAl was obtained from Sherritt-Gordon

Mines Ltd. in the form of pressed and sintered billets with a nominal diameter of 7.6 centimeters.

Unrecrystallized DS-NiCr was heat-treated before testing. The alloys in billet form were subjected to various thermomechanical processing and heat-treatment schedules in order to obtain an elongated microstructure with a uniform large grain size. The final processing and heat-treatment schedules for all of the ODS alloys are shown in table II. The average grain-size parameters determined by lineal analysis and the crystallog-raphic textures of the alloys are given in table III. Dispersoid particle sizes or interparticle spacings were not determined for these alloys.

Round-bar tensile type specimens with a nominal 2.8-centimeter-long by 0.51-centimeter-diameter gage section were centerless ground from the heat-treated alloys. In general, tapered grip end specimens were used; however, threaded grip end specimens were used for DS-NiCr tested in the long transverse direction and for Inconel MA-754 in order to conserve material. All alloys were tested parallel to the extrusion axis. In addition, DS-NiCr and Inconel MA-754 were tested in the long transverse direction (long axis of cross section of bar).

All creep testing was conducted in accordance with ASTM specifications at 1365 K in air on constant-load test machines. Creep tests were generally designed to produce between 0.1 and 1 percent strain in 100 hours; however, a few specimens were tested to rupture. Elongation as a function of time was measured optically from scribed platinum strips which had been spot-welded to the shoulders of the test specimens. Testing was interrupted for many specimens after 100 to 150 hours; these specimens were then subjected to tensile testing to determine the effect of prior creep. The results of residual tensile testing are described in reference 20.

In addition to creep tests, tensile tests were conducted at room temperature and at 1365 K. Room-temperature testing was conducted on an oil hydraulic test machine at a crosshead speed of about 0.001 centimeter per second, with strain through the 0.2-percent yield measured by a clip-on extensometer. Tensile testing at 1365 K was conducted at a constant crosshead speed of 0.002 centimeter per second, and the 0.2-percent yield stress was estimated from plots of load as a function of crosshead motion. Selected tensile and rupture tested specimens were subjected to metallographic examination.

RESULTS AND DISCUSSION

Tensile Properties

The tensile properties of the ODS alloys at room temperature and at 1365 K are shown in table IV. At room temperature, the Ni-16Cr-Al type alloys are considerably

stronger than the Ni or Ni-20Cr type; this strength is probably due to the presence of γ' and solid-solution hardening effects of Al. The higher strength of Inconel MA-754 in comparison to DS-NiCr is probably due to the combined solid-solution hardening effects of Al and Ti. Both Inconel MA-754 and DS-NiCr are stronger parallel to the extrusion axis ([100] direction) than in the long transverse direction ([011] direction). This is probably due to slip on multiple slip systems leading to strain hardening for the [100] direction, while only one slip system is operating in the [011] direction.

At room temperature, all the alloys exhibited reasonable tensile elongations, and all failures were intragranular. DS-NiCr tested in the long transverse direction exhibited multiple necks which lead to the very high tensile elongation.

With the exception of Inconel MA-754, all of the alloys possessed similar tensile strengths at 1365 K; Inconel MA-754 is considerably stronger. DS-NiCr and TD-NiCrAl were very ductile, and the fractures were intragranular. Inconel MA-754 and DST-NiCrAl tested parallel to the extrusion axis exhibited reasonable ductility; however, the fractures were intergranular, with considerable grain boundary cracking evident in the microstructures. Inconel MA-754 tested in the long transverse direction exhibited low ductility, and the fracture was intergranular. TD-Ni possessed moderate ductility, and the fracture was probably intergranular.

The tensile test data in table IV for TD-Ni, DS-NiCr, and TD-NiCrAl agree well with the data of other investigators (refs. 1 and 9).

Comparison of the 1365-K tensile-strength data in table IV with the grain-size parameters in table III reveals that no correlation exists between strength and grain size. However, there does seem to be a linear relationship, as suggested by Wilcox and Clauer (ref. 12), between 1365-K tensile-strength properties and grain aspect ratio for large-grain-size ODS alloys tested parallel to the extrusion axis. This relationship is shown in figure 1; it should be noted that the one example of a very low grain aspect ratio material (Inconel MA-754 transverse) does not follow the commonly held concept of low grain aspect ratio being equivalent to low strength. Additionally, the strength of TD-Ni is not predictable by extrapolation of the curve in figure 1. This is probably a result of the small grain size of TD-Ni and the previously observed behavior (ref. 12) that ODS-Ni alloys are weaker than ODS-Ni base alloys.

Creep Behavior

<u>Creep data and analysis.</u> - Table V contains the creep data tabulated for all the alloy-direction combinations evaluated in this study. Data are presented for strains at 1 hour to indicate the amount of transient creep; in general, the alloys were in steady-state creep after 1 hour of testing. With the exception of DS-NiCr, the alloys were generally in third-stage creep after about 0.5 percent strain. DS-NiCr tested parallel to the

extrusion axis did not enter third-stage creep until about 2 percent strain. DS-NiCr tested in the long transverse direction appeared to undergo very little deformation prior to failure; however, measurements of the elongation and reduction in area in the failed specimens indicated that considerable deformation occurred in a short time during the rupture process. Because of this and the scatter in the creep-rupture data in table V, the creep behavior of DS-NiCr in the long transverse direction could not be characterized. In this study, creep rates less than 10^{-9} per second could not be determined, as they were below the limit of detection.

With the exception of DS-NiCr tested parallel to the long transverse direction, the alloys exhibited the usual empirical creep-rate relationship for stress dependency at strain rates greater than about 2×10^{-9} per second:

$$\dot{\epsilon} \propto \sigma^{\eta}$$
 (1)

where $\dot{\epsilon}$ is the strain rate, σ is the applied tensile stress, and η is the stress exponent. The stress exponents for the alloys are listed in table VI; in general, the power-law exponents are high, which is apparently normal for most ODS alloys (ref. 18).

All of these alloy systems appear to possess a ''threshold'' stress below which creep does not occur. Approximate threshold stresses are given in table VI. These were estimated from the strain-rate data and the shape of strain-time curves (obtained from table V), and the residual tensile property data (ref. 20). In general, creep exposures at or slightly below the threshold stress did produce a small amount (≈ 0.1 percent) of transient creep. Once transient creep was finished, however, test specimens exhibited little, if any, further deformation over extended periods of time (e.g., specimens N-10, L-14, T-5, S-8, ST-4, TL-12, and T-9-5 in table V). The strain-rate data were also fitted to a modified power-law stress dependency equation:

$$\dot{\epsilon} \propto (\sigma - \sigma_0)^{\eta'}$$
 (2)

where σ_0 is the estimated threshold stress for creep from table VI, and η^* is the effective stress exponent. The effective stress exponents are also presented in table VI. Figure 2 illustrates a typical example of the modified power-law behavior. In general, the fit of the strain-rate data for the polycrystalline alloys is fair, at best. However, all these alloys possessed low (less than 3) effective stress exponents, which suggests that diffusional creep is a possible mechanism.

The creep rate of DS-NiCr tested parallel to the extrusion axis was well described by equation (2) for $\dot{\epsilon} > 10^{-8}$ per second, where the effective stress exponent was about 9. An effective stress exponent of 9 is in agreement with Lin and Sherby's analysis (ref. 19) of Lund and Nix's creep data (ref. 18) for single-crystal Ni-20Cr-2ThO₂ (TD-NiCr).

Metallography. - Rupture-tested TD-Ni possessed a few cavities in the vicinity of the fracture, and DS-NiCr tested parallel to the extrusion axis to rupture contained a few small cracks within and on the surface of the necked region. The microstructure of the interrupted-creep-test specimens after room-temperature tensile testing was identical to the as-received (untested) microstructure for these two alloy-direction combinations (ref. 20).

Metallography of the failed specimens of Inconel MA-754 and TD-NiCrAl tested parallel to the extrusion axis and DS-NiCr tested in the long transverse direction revealed that the fractures were intergranular, and it also showed evidence of diffusional creep. Such evidence consisted of dispersoid-free bands, intergranular cavitation and/or cracking, and internal oxidation at or near grain boundaries. Typical examples of dispersoid-free bands and cracking coupled with internal oxidation are shown in figure 3. Similar microstructural features were seen in many interrupted-creep-test specimens of Inconel MA-754, TD-NiCrAl, and DST-NiCrAl after tensile testing at room temperature (ref. 20).

While such features are clearly visible in the gage sections of test specimens which experienced as little as 0.2 to 0.3 percent creep strain, these microstructural features are not uniformly distributed throughout the gage section; in fact, the bulk of the microstructure is identical to that of as-received (untested) alloy. In essence, the microstructure of tested specimens of Inconel MA-754, TD-NiCrAl, and DST-NiCrAl, and DS-NiCr tested in the long transverse direction indicates that creep (or at least that part due to diffusional creep mechanisms) is not homogeneous. Similar behavior has been observed in creep-tested and stress-rupture-tested TD-NiCr sheet (ref. 16). Additionally, Lund and Nix (ref. 18), after studying creep in Ni-20Cr-2ThO₂ single crystals, concluded that creep in polycrystalline ODS alloys is probably an inhomogeneous process. If creep in polycrystalline alloys is truly inhomogeneous, then correlations between strain rate and stress (e.g., eqs. (1) and (2)) may not be meaningful.

Threshold stress. - As previously indicated, it appears that all alloy-direction combinations tested in this study have a threshold stress for creep. For DS-NiCr tested parallel to the extrusion axis, the threshold stress is associated with some type of normal dislocation motion or generation, since DS-NiCr tested parallel to the extrusion axis is essentially a perfect single crystal (no transverse boundaries). Recently, Lund and Nix (ref. 18) reported creep-test results for single-crystal Ni-20Cr-2ThO₂ (TD-NiCr) produced by directional recrystallization; they also found an apparent threshold stress for creep and suggest that it is the Orowan stress. At 1365 K, their threshold stress was about 79 megapascals, which agrees well with the 69 megapascals measured for DS-NiCr in this study.

With the exception of DS-NiCr tested parallel to the extrusion axis, all the ODS alloys tested in this study possessed transverse boundaries in the test section. Thus, these alloys are capable of undergoing slow-strain-rate plastic deformation by diffusional

the stress required to produce a strain rate of 2.78×10⁻⁸ per second (1 percent creep in 100 hr) by diffusional creep at 1365 K for a 250-micrometer-grain-size ODS Ni alloy is about 17 megapascals. Both Inconel MA-754 and TD-NiCrAl have approximately a 250-micrometer grain size; and, as can be seen in table V, both alloys require stress levels in excess of 17 megapascals in order to produce a strain rate of 2.8×10⁻⁸ per second. Thus, diffusional creep mechanisms alone can produce more deformation than is observed. These calculations and the microstructural evidence of diffusional creep (dispersoid-free bands) in large-grain-size ODS alloys support the concept that the threshold stress is associated with diffusional creep rather than dislocation creep mechanisms.

Explanation of the threshold stress in TD-Ni is more difficult than those for single-crystal or large-grain-size alloys. It is tempting to also ascribe this threshold stress to diffusional creep; however, because of the very small grain size in TD-Ni, exceeding the threshold stress by moderate amounts should result in extremely high diffusional creep rates. For example, at 1365 K, exceeding the threshold stress by 1 megapascal would mean a steady-state diffusional creep rate of about 10^{-4} per second. Accounting for the high grain aspect ratio of TD-Ni would lower these creep rates; however, Raj and Ashby (ref. 15) estimate that the rate would only be reduced by the square root of the grain aspect ratio ($\sqrt{19}$ for TD-Ni). Either creep in TD-Ni is not controlled by diffusional mechanisms, or perhaps the measured threshold stress is at the lower end of a range of threshold stresses. Such a range could account for inhomogeneous deformation resulting in the low strain rates.

The threshold stress seems to be associated with diffusional creep in the large-grain-size ODS alloys: Inconel MA-754, TD-NiCrAl, and DST-NiCrAl. Comparison of the threshold stress values in table VI with the grain-size parameters in table III gives no indication of any relationship between threshold stress and grain size. There does, however, appear to be a linear relationship between 1365 K threshold stresses and grain aspect ratio, as shown in figure 4. Included in this figure are the threshold-stress data for thin TD-NiCr sheet (ref. 17); the pertinent data for this material are presented in table VII. The curve shown in figure 4 is described by

$$\sigma_0 = 6 + 18.1 \text{ (GAR)}$$
 (3)

where σ_0 is in megapascals, and GAR is the grain aspect ratio reported in tables III and VII. This figure indicates that the threshold stress for creep in polycrystalline alloys is dependent on the ratio of grain boundary area in shear to grain boundary area in tension. While equation (3) accurately describes the relationship between threshold stress and grain aspect ratio for thick material, as can be seen in table VII, equation (3) overestimates the threshold stress for very thin material. However, as the 0.025-

centimeter-thick TD-NiCr sheet was consistently weaker than the 0.051-centimeter-thick sheet (ref. 17), it is not surprising that the 0.025-centimeter-thick sheet did not show results consistent with equation (3). Apparently, 0.025-centimeter-thick TD-NiCr sheet is not representative of bulk material.

Overall, it appears that the threshold stresses for ODS Ni-20Cr and Ni-16Cr-Al type alloys are uniquely dependent on the grain aspect ratio. For such alloys with grain sizes between 150 and 400 micrometers and grain aspect ratios between 0.8 and 3.5, threshold stresses at 1365 K can be calculated from equation (3) with, apparently, good accuracy. Use of equation (3) beyond the stated limits of grain size and grain aspect ratio is not recommended without supporting experimental data. For instance, at sufficiently high grain aspect ratios, the predicted threshold stress would exceed the stress required for dislocation motion (Orowan stress).

B)

As shown in references 17 and 20, the residual room-temperature tensile properties of large-grain-size ODS alloys are greatly affected by prior elevated-temperature creep. For example, prior creep strains as low as 0.2 percent can reduce the tensile ductility as much as 50 percent. Such behavior seemingly limits the use of large-grain-size ODS alloys to conditions where creep does not occur. Thus, threshold stress may be the most important creep-strength property for these alloys. For use of these particular alloys, the threshold stress is recommended as the base design criterion, rather than the more conventional criterion of a stress to produce a known amount of creep in finite time.

Finally, it should be noted that the threshold stresses estimated in this work are based on the results of creep tests of nominally 100 hours at 1365 K. It was shown in references 17 and 18 that threshold stresses are dependent on temperature; in addition, threshold stresses may be dependent on time. Longer creep tests (over 100 hr) increase the ability to measure lower creep rates (less than 10⁻⁹ per second). Thus, threshold stresses estimated from creep curves may be reduced, since one is better able to detect very small strain rates.

CONCLUSIONS

Based on a study of tensile creep at 1365 K of several ODS Ni-base alloys, the following conclusions are drawn:

- 1. ODS alloys possess threshold stresses for creep.
- 2. Creep in polycrystalline ODS alloys is an inhomogeneous process.
- 3. The threshold stresses in large-grain-size ODS Ni-20Cr and Ni-16Cr-Al type alloys are dependent on the grain aspect ratio.

4. Maximum design criteria for large-grain-size ODS alloys should be limited to the threshold stress for creep.

Lewis Research Center,

National Aeronautics and Space Administration, Cleveland, Ohio, December 28, 1976, 505-01.

REFERENCES

- 1. Doble, G. S.; and Quigg, R. J.: Effect of Deformation on the Strength and Stability of TD-Nickel. Trans. Metall. Soc. AIME, vol. 233, no. 2, Feb. 1965, pp. 410-415.
- 2. Wilcox, B. A.; and Clauer, A. H.: Creep of Thoriated Nickel Above and Below 0.5 Tm. Trans. Metall. Soc. AIME, vol. 236, no. 4, Apr. 1966, pp. 570-580.
- 3. Lowell, Carl E.; Grisaffe, Salvatore J.; and Deadmore, Daniel L.: Oxidation of TD Nickel at 1050° and 1200° C as Compared with Three Grades of Nickel of Different Purity. Oxid. Met., vol. 4, no. 2, June 1972, pp. 91-111.
- Wilcox, B. A.; Clauer, A. H.; and McCain, W. S.: Creep and Creep Fracture of Ni-20Cr-2ThO₂ Alloy. Trans. Metall. Soc. AIME, vol. 239, no. 11, Nov. 1967, pp. 1791-1795.
- 5. Fritz, L. J.; Koster, W. P.; and Taylor, R. E.: Characterization of the Mechanical and Physical Properties of TD-NiCr (Ni-20Cr-2ThO₂) Alloy Sheet. (Metcut Research Assoc.; NAS3-15558) NASA CR-121221, 1973.
- 6. Lowell, Carl E.; et al.: Oxidation of Ni-20Cr-2ThO $_2$ and Ni-30Cr-1.5Si at 800° , 1000° , and 1200° C. NASA TN D-6290, 1971.
- Johnston, James R.; and Ashbrook, Richard L.: Oxidation and Thermal Fatigue Cracking of Nickel- and Cobalt-Base Alloys in a High Velocity Gas Stream. NASA TN D-5376, 1969.
- 8. Lowell, Carl E.; and Sanders, William A.: Mach 1 Oxidation of Thoriated Nickel Chromium Alloys at 1204° C (2200° F). Oxid. Met., vol. 5, no. 3, Dec. 1972, pp. 221-239.
- Klingler, L. J.; et al.: Development of Dispersion Strengthened Nickel-Chromium Alloy (Ni-Cr-ThO₂) Sheet for Space Shuttle Vehicles, Part 2. (Fansteel, Inc.; NAS3-13490) NASA CR-121164, 1972.

- 10. Klarstrom, D. L.; and Grierson, R.: Optimization of an Oxide Dispersion Strengthened Ni-Cr-Al Alloy for Gas Turbine Engine Vanes. (Cabot Corp.; NAS3-17806) NASA CR-134901, 1975.
- 11. Deadmore, Daniel L.; Lowell, Carl E.; and Santoro, Gilbert J.: High Gas Velocity Oxidation and Hot Corrosion Testing of Oxide-Dispersion-Strengthened Nickel-Base Alloys. NASA TM X-71835, 1975.
- 12. Wilcox, B. A.; and Clauer, A. H.: Dispersion Strengthening. The Superalloys. C. T. Sims and W. C. Nagel, eds., John Wiley & Sons, 1972, pp. 197-230.
- 13. Bailey, P. G.: Oxide Dispersion Strengthened Alloys for Aircraft Turbine Engine Vanes. Proceedings of the Sixth National Technical Conference of Materials on the Move. Soc. Advancement Mater. Proc. Eng., 1974, pp. 208-217.
- 14. Kane, R. D.; and Ebert, L. J.: Creep Deformation of TD-Nickel Chromium. Metall. Trans., vol. 7A, no. 1, Jan. 1976, pp. 133-137.
- 15. Raj, R.; and Ashby, M. F.: On Grain Boundary Sliding and Diffusional Creep. Metall. Trans., vol. 2, no. 4, Apr. 1971, pp. 1113-1127.
- 16. Whittenberger, John D.: Diffusional Creep and Creep-Degradation in Dispersion-Strengthened Ni-Cr Base Alloys. Metall. Trans., vol. 4, no. 6, June 1973, pp. 1475-1483.
- 17. Whittenberger, John D.: Observations on the Relationship of Structure to the Mechanical Properties of Thin TD-NiCr Sheet. Metall. Trans., vol. 7A, no. 5, May 1976, pp. 611-619.
- 18. Lund, R. W.; and Nix, W. D.: High Temperature Creep of Ni-20Cr-2ThO₂ Single Crystals. Acta Metall., vol. 24, no. 5, May 1976, pp. 469-481.
- 19. Lin, J.; and Sherby, O. D.: Mechanical Behavior of Dispersion Hardened Materials at Elevated Temperature. 2nd Annual Report, Stanford Univ. (NASA Grant NGR-05-020-671), 1975.
- 20. Whittenberger, J. Daniel: Creep Degradation in Oxide-Dispersion-Strengthened Alloys. NASA TN D-8421, 1977.

TABLE I. - NOMINAL COMPOSITIONS OF ODS

ALLOYS TESTED

Alloy	Composition, wt %					
	Cr	Al	С	ThO ₂	Y2O3	Ni
TD-Ni (heat 3062)			0.027	2		Bal.
DS-NiCr	20. 3		. 009	2		Bal.
Inconel MA-754 ^a (heat 0T0055B)	20	0.6	. 07	-	0.6	Bal.
TD-NiCrAl (heat 3939)	16	4.2	. 05	2		Bal.
DST-NiCrAl	16	5	. 013	2		Bal.

^aAlso contains 1.5 Fe and 1.0 Ti.

TABLE II. - FINAL THERMAL-MECHANICAL PROCESSING AND HEAT-

TREATMENT SCHEDULES FOR ODS ALLOYS

Alloy	Thermal-mechanical processing	Heat treatment
TD-Ni	(a)	(a)
DS-NiCr	Extruded approx. 12:1 at approx. 1365 K by vendor	Heated from 1475 K
		to 1590 K in 2 hr
Inconel MA-754	(a)	(a)
TD-NiCrAl	Extruded 12:1 at 1365 K to 3-by 10-cm sheet bar;	2 hr at 1640 K
	hot rolled to 1.5-cm thickness in two 30-percent	
	passes at 1365 K	
DST-NiCrAl	Canned in steel; extruded 16:1 at 1395 K to approx.	2 hr at 1640 K
	1.7-cm diam bar	

^aNot reported by vendor.

TABLE III. - GRAIN-SIZE PARAMETERS AND TEXTURE OF ODS ALLOYS

Alloy	Charac	eteristic leng	th, μm	Average	Grain aspect	Orientation
	Parallel to extrusion axis, ^L 1	In long transverse direction,	In short transverse direction, L ₃	grain size, $0.85 \sqrt[3]{L_1 L_2 L_3},$ μm	ratio, GAR, $L_1/\sqrt[4]{L_2L_3}$	
TD-Ni	25	1.3	diam	3.5	19	Wire; [100] parallel to extrusion axis
DS-NiCr	Single crystal with very large elongated low-angle grains approximately 1 cm diameter by 10 cm long. Low-angle grains elongated along extrusion axis.					$[100]$ parallel to extrusion axis $[011]$ parallel to long transverse direction $[0\overline{1}1]$ parallel to short transverse direction
Inconel MA-754	530	180	115	250	3.68 ^a 0.73	Same as DS-NiCr
TD-NiCrAl	490	285	150	235	2.37	Same as DS-NiCr
DST-NiCrAl	1200	1200 300 diam (duplex: 660 and 120 μm diam)		400	b _{1.82}	Wire; [522] parallel to extrusion axis

^aCalculated from $\sqrt{L_2L_3}/L_1$ for transverse testing. ^bCalculated for larger grain diameter.

TABLE IV. - TENSILE PROPERTIES OF SEVERAL ODS ALLOYS

Alloy	Test direction	Tensile properties							
		0.02-Percent yield stress, MPa	0.2-Percent yield stress, MPa	Ultimate tensile strength, MPa	Elongation, percent	Reduction in cross-sectional area, percent			
		Room temp	erature						
TD-Ni	Parallel to extrusion axis	440	469	482	20	79			
		350 373	418 435	493 490	19 19	82 78			
DS-NiCr	Parallel to extrusion axis	373	398	803	22	28			
		352	380	789	26	36			
	Long transverse		442	626	42	43			
Inconel MA-754	Parallel to extrusion axis	626	688	1052	23	21			
		589	646	1021	20	23			
	Long transverse	581	655	932	23	29			
		571	657	937	23	27			
TD-NiCrAl	Parallel to extrusion axis	730	786	1179	11	10			
DST-NiCrAl	Parallel to extrusion axis	675	746	1062	16	14			
		677	777	1128	15	16			
		692	720	1073	13. 6	14			
		1365	К —						
TD-Ni	Parallel to extrusion axis		104	119	3.6	6			
			99	112	4.5	6			
			93	120	3. 6	6			
DS-NiCr	Parallel to extrusion axis		94	107	17	69			
			97	106	19	52			
Inconel MA-754	Parallel to extrusion axis		148	151	9.5	12			
			149	152	9.5	11.3			
	Long transverse	-	154	154	2.6	1.5			
يم سد		- 	154	154	2.5	1.6			
TD-NiCrAl	Parallel to extrusion axis		102	108	21	45			
			99	105	19	44			
DST-NiCrAl	Parallel to extrusion axis		90	103	12	15			
			83	92	7	11			
			81	92	13. 6	13			

TABLE V. - CREEP AND CREEP RUPTURE PROPERTIES OF SEVERAL ODS ALLOYS AT 1365 K

Specimen	Stress,	Time,	Stra	in, percent	Steady-state	Comments
	M Pa	hr	After 1 hr	At end of test	creep rate per second	
			TD-N	i; parallel to ext	rusion axis	
a _{N-1}	75.8	91. 1 (failed)	0.21	≈2.5	1.3×10 ⁻⁸	0, 65 Percent strain at 79, 1 hr
a _{N-6}	68,9	43.3 (failed)	.4	≈2	1.45×10 ⁻⁸	0, 74 Percent strain at 43, 25 hr
N-50	65.5	142.4	.27	. 49	2.35×10 ⁻⁹	
N-53	62	120. 5	. 12	. 35	Ill-defined curve	
N-52	58.6	141.0	. 16	. 30	1.6×10 ⁻⁹	
N~56	55, 1	148.4	.05	. 20	<10 ⁻⁹	
^a N-5	55.1	116.9	. 14	. 18	<10 ⁻⁹	Curve flat after 1 hr
N-10	48.2	117. 3	. 19	. 25	<10-9	Curve flat after 20 hr
a _{N-4}	48.2	120. 5	. 02	. 12	<10 ⁻⁹	Curve flat after 20 hr
			Inconel M	A-754; parallel	to extrusion axis	
L-16	89.6	73. 1	0,2	1.0	2.5×10 ⁻⁸	
L-13	86.1	82, 5	. 15	≈.5	1.2×10 ⁻⁸	0.48 Percent strain at 74.7 hr;
						pulled out grip at 82,5 hr
L-2	82.7	100, 1 (failed)	.1	≈3.5	1×10-8	0.72 Percent strain at 88.7 hr
L-5	82,7	100, 4	.1	.435	1 1×10-8	8, 13 1 02 00.00 00.00 00.00
L-12	82.7	69. 0	. 15	≈.3	7×10 ⁻⁹	0,27 Percent strain at 50.5 hr:
						pulled out grip at 69.0 hr
L-4	82.7	67. 8 (failed)	. 08	≈3,5	2,5×10 ⁻⁸	1.10 Percent strain at 67.2 hr
L-7	75.9	141. 1	. 05	. 313	4.3×10 ⁻⁹	
L-15	75.9	47.8	. 12	≈, 22	4.9×10 ⁻⁹	0, 17 Percent strain at 20, 5 hr
						pulled out grip at 47, 8 hr
L-14	72.3	141.9	. 10	. 11	<10 ⁻⁹	Curve flat after 1 hr
L-8	68.9	141.6	. 08	. 1	<10 ⁻⁹	Curve flat after 1 hr
L-3	65.5	145, 1	. 05	. 12	<10 ⁻⁹	Curve flat after 20 hr
	,		Inconel M	A-754; long tran	sverse direction	
T-7	34.5	67. 3	0.08	1,67	3.8×10 ⁻⁸	
T-6	27.6	72. 7	. 15	1.24	3.35×10 ⁻⁸	
T-1	24.1	101.0	. 04	.91	1. 1×10 ⁻⁸	
T-13	22.4	94. 1	. 11	≈.22	3.35×10 ⁻⁹	0, 22 Percent strain at 90, 8 hr
			1	1	1	power failure at 94, 1 hr
T-3	20.7	120. 3	0	49	1, 3×10 ⁻⁸	
T-11	20.7	149. 2	0	. 06	1×10 ⁻⁹	
T-2	18.9	148. 7	. 03	.44	4.45×10 ⁻⁹	
T-14	17.2	149.5	. 02	. 12	<10-9	Curve flat after ≈50 hr
T-5	13.8	148, 3	. 05	0	<10-9	Curve flat

			DS-Nic	Cr; parallel to extra	ision axis	
S-11	86.1	3.05 (failed)	2.2	17.3 (61 percent	4.4×10 ⁻⁶	5.04 Percent strain at 2.4 hr
				reduction in area)		
S-15	82.1	11.9 (failed)	.085	17.3 (60 percent	8.6×10 ⁻⁷	2.97 Percent strain at 7.25 hr
S-3	79.2	98. 3	. 5	reduction in area)	2.6×10 ⁻⁸	
S-5	75.9	115. 1	0	.6	1. 1×10 ⁻⁸	
S-9	72.3	101.4	0	.3	7. 6×10 ⁻⁹	
S-8	68.9	500	.2	.2	<10 ⁻⁹	Curve flat after 1 hr
S-12	68.9	115. 7	. 03	,08	<10-9	<u> </u>
S-13	65.5	101. 0	. 03	. 18	4×10 ⁻⁹	
S-14	62.0	119.5	. 02	. 13	<10 ⁻⁹	Curve flat after 7 hr
	·	<u> </u>	DS-Ni	Cr; long transverse	direction	
ST-11	82.7	88, 6	0.07	0, 32	5.2×10 ⁻⁹	
ST-12	79.2	31, 5 (failed)	. 02	5.4 (13 percent re-	<10 ⁻⁹	0.11 Percent strain at 31.4 hr;
		V-(V		duction in area)		curve flat after 3, 5 hr
ST-6	79.2	17.3 (failed)	.1	5. 7 (6. 2 percent re	<i>-</i>	0.16 Percent strain at 5.7 hr
				duction in area)		
ST-1	75.9	113, 7 (failed)	. 04	3, 8 (9, 3 percent re	-<10 ⁻⁹	0.08 Percent strain at 104.2 hr
				duction in area)	1	curve flat after 3,5 hr
ST-8	75.9	. 5 (failed)		3 (3, 5 percent re-		0.03 Percent strain at 0.4 hr
	1			duction in area)		
ST-9	68.9	146, 2	. 01	. 1	1.5×10 ⁻⁹	
ST-7	62.0	150. 3	. 06	, 15	2×10 ⁻⁹	
ST-4	27.6	147. 9	. 03	. 03	<10 ⁻⁹	Curve flat after 1 hr
	ased to 55,		. 03	. 07	<10 ⁻⁹	Curve flat after 10 hr
ST-5	24.1	141, 1	0	0	<10-9	Curve flat
	ased to 48.		. 01	. 03	<10-9	Curve flat after 5 hr
ST-3	34.5	148. 6	0	0	<10 ⁻⁹	Curve flat
ST-2	31.0	150. 3	ō	0	<10 ⁻⁹	Curve flat
			TD-NiC	CrAl; parallel to ext	rusion axis	
TL-5	75. 8	39. 5 (failed)	0.2	7		2.4 Percent strain at 28.6 hr
TL-4	68.9	<46. 9 (failed)	.2	5. 4	1×10 ⁻⁷	1 Percent strain at 23, 1 hr
TL-11	62.0	90. 0 (failed)	,1	Multiple fracture	1.9×10 ⁻⁸	0.94 Percent strain at 68.6 hr
TL-9	55.1	74. 8	. 07	1, 8	2.6×10 ⁻⁸	o. of a creek seam at oo. o in
TL-7	55,1	44.0	. 15	.57	2.35×10 ⁻⁸	1
TL-12	51, 7	115. 7	. 1	.11	<10-9	Curve flat after 1 hr
TL-10	48, 2	118.0	. 05	. 12	<10-9	Curve flat after 5 hr
TL-6	44,8	115. 8	. 05	. 05	<10-9	Curve flat after 1 hr
TL-8	41.3	114. 2	. 02	.1	<10-9	our ve hat after 1 in
			DST-Nie	CrAl; parallel to ex	trusion axis	
T-8-8	62.0	102.0	0.55	3,46		Steady state region not defined
T-8-6	55, 1	142.2	. 08	. 68	7.3×10 ⁻⁹	,
T-9-3	55.1	137. 9	. 01	. 51	1,65×10 ⁻⁸	Essentially no creep first 50 hr
T-9-2	51.7	163. 9	. 15	.3	3,40×10 ⁻⁹	• • • • • • • • • • • • • • • • • • • •
T-9-4	48,2	118. 9	0	. 19	2.85×10 ⁻⁹	
T-8-7	48.2	140.3	. 05	. 28	4.2×10 ⁻⁹	
T-8-12	44.8	168. 0	. 11	≈.3	1.75×10 ⁻⁹	
T-9-7	44.8	165. 8	. 05	, 25	1.85×10 ⁻⁹	
T-8-4	41.3	148.9	. 08	. 24	<10 ⁻⁹	Curve flat after 75 hr
T-9-5	41.3	147. 8	0	0	<10-9	Curve flat
T-8-3	41.3	150. 1	0	. 18	2.4×10 ⁻⁹	
T-9-6	41.3	146. 8	. 03	. 27	3.7×10 ⁻⁹	1

aPlasma coated with Ni-20Cr, approximately 100 μm thick.

TABLE VI. - STRESS EXPONENTS AND ESTIMATED THRESHOLD STRESSES FOR ODS ALLOYS AT 1365 K

Alloy	Direction	Stress	Threshold	Effective
ĺ		exponent,	stress,	stress
		η	σ ₀ , MPa	exponent,
TD-Ni	Parallel to extrusion axis	13.5	53	2.4
Inconel MA-754	Parallel to extrusion axis	18	72	1
Inconel MA-754	Long transverse	16	17	1.4
DS-NiCr	Parallel to extrusion axis	41	69	9
DS-NiCr	Long transverse	(a)	55	(a)
TD-NiCrAl	Parallel to extrusion axis	23	52	(a)
DST-NiCrAl	Parallel to extrusion axis	10.5	41	1

^aCould not be determined.

TABLE VII. - GRAIN-SIZE PARAMETERS AND THRESHOLD STRESS

FOR THIN SHEET OF TD-NiCr (Ni-20Cr-2ThO $_2$)

[From ref. 17.]

Heat	Orientation	, m			Grain size,	Grain	Threshold stress, MPa	
	relative to sheet rolling direction	Parallel to test direction,	Transverse to test direction,	In sheet- thickness direction,	$0.85 \sqrt[3]{L_1 L_2 L_3},$ μm	aspect ratio, $L_1/\sqrt{L_2L_3}$	Measured	Calculated by equation (3)
		L ₁	$^{ extsf{L}_2}$	$^{\mathrm{L}_3}$				
3636 (0.051	Parallel	290	230	50	150	2.7	a ₅₉	55. 0
em thick)	Normal	230	290	50	150	1.9	38	40.5
3712 (0.051	Parallel	640	360	100	240	3.37	65	67.0
cm thick)	Normal	360	640	100	240	1.42	34	31.7
3637 (0.025	Parallel	390	290	53	165	3.15	45	63.0
cm thick)	Normal	290	390	53	165	2.0	32	42.2

a_{Estimated}.

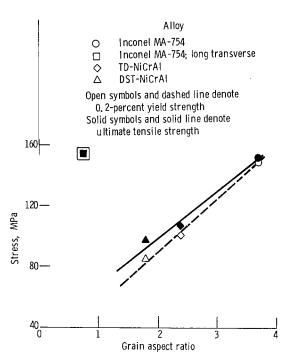


Figure 1. - Tensile strength as a function of grain aspect ratio for several large grain size ODS alloys at 1365 K.

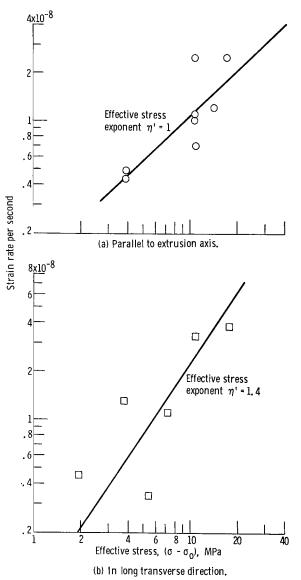
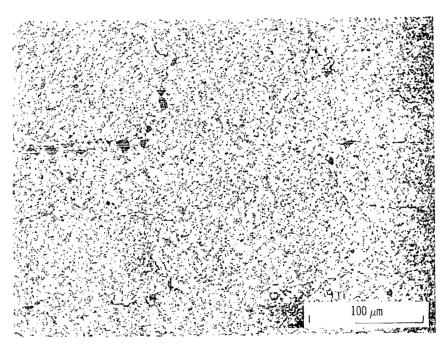


Figure 2, - Steady state creep rate of Inconel MA-754 as a function of the effective stress,



(a) Dispersoid-free bands in Inconel MA-754 tested at 82.7 megapascals for 100.4 hours. Specimen electrolytically stain etched with chromic acid mixture (100 cm 3 H₂O, 10 cm 3 H₂SO₄, 2 g chromic acid) at 3 to 5 volts.



(b) Cracking and internal oxidation in TD-NiCrAl tested at 62 megapascals for 90 hours; multiple fracture. Specimen electrolytically etched with buffered aqua regia (2 parts by volume aqua regia, 1 part glycerine) at 3 to 5 volts.

Figure 3. - Typical microstructures of ODS alloys creep rupture tested at 1365 K.

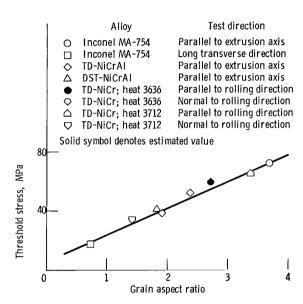


Figure 4. - Threshold stress as a function of grain aspect ratio for large grain size ODS alloys at 1365 K.

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